Effect of Cold Rolling sequence on the Texture Development and Magnetic Loss of Grain Oriented Electrical Steels

Mehdi Salari1*

1Department of Metallurgical Engineering, Sirjan Branch, Islamic Azad University, Sirjan, Iran
*E-mail of corresponding author: mehsalari@gmail.com
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Abstract
Electrical steels are important alloys widely used in electrical instruments. The magnetic properties and low core loss of grain oriented silicon steels are closely related to the sharpness of Goss texture (\{110\}<001>). The direction of <001> is the softest direction for magnetism. In this work, the microstructure and dependence of orientation density along the major texture fibers on the applied cold rolling sequences in the CRGO samples were investigated. Variation of the hysteresis losses revealed that hysteresis losses were high for the specimen treated at higher secondary cold reduction. In this sample, the conditions for selective growth of Goss oriented grains are violated which leads to development of grains with random orientations almost equally. The results also indicated that the annealing of the cold rolled specimen at the higher first cold reduction and lower secondary reduction showed increase of the \{110\}<001>texture component intensity and drastically decrease in hysteresis losses.

Keywords
Magnetic Steels, Grain Oriented, Texture Evolution, Goss texture, Hysteresis Losses.

1. Introduction
Silicon steels are soft magnetic materials widely used for power, distribution transformer and large generators cores. The magnetic properties, low core loss and high permeability of these steels are closely related to the sharpness of \{110\}<001> or so-called Goss texture, because the [001] is the easiest axis of magnetization and demagnetization[1].

Production of electrical steel process includes steelmaking, casting, annealing, hot rolling, normalizing annealing, cold rolling, first recrystallization annealing, secondary recrystallization annealing, and heat flattening coating [2].

The strong Goss texture of grain-oriented silicon steel is the result of a complex processing scheme. This texture develops due to a discontinuous or abnormal Goss grain growth during a high-temperature annealing at the end of the industrial procedures [3].

Two aims of cold rolling in production of electrical steels are reduction of final thickness for lower magnetic loss and further driving force for nucleation during annealing step and finer structure. The sites that have more deformation and disorientation are suitable for nucleation of recrystallized grains [3-5].

Matsuo [6] pointed out that in cold strains (90%) compared with lower strains (50 to 65%) the amount of Goss texture and intensity of \{111\}<112>orientation are high. The reason was narrower shear bands that produced during cold rolling.
Harase et al. [7] concluded that with increasing strain, the number of suitable sites for nucleation increases and by reduction of the space required for grain growth, recrystallization grain diameter decreases. They emphasized that the rapid growth of the Goss grains in the secondary recrystallization occurs when grain size of the primary recrystallization be small. Thus, cold rolling has a great impact on the success or lack there of in the production of the Goss texture. Many researchers [8-11] have stressed that the shear bands are the most important sites for nucleation of the Goss area during recrystallization. Hutchinson [9] clarified the most important factors, as the formation of shear bands, the temperature of the rolls, the friction and lubrication, the stress and strain situation, local in homogeneity in grain size, size of precipitates and fault in one area of the sample. Heo[12] investigated the lubricating effect on cold-rolled magnetic steels and concluded that in the final texture of the un-lubricated sample, the main orientation was{110}<001>, and in the sample cold rolled with lubricants, the components of{554}<225>, {100}<011> and{110}<001> had greater presence. He also investigated the cold rolling and heating rate effects on magnetic properties and texture of electrical steels and concluded that in the samples cold rolled without lubricant, the Goss texture was strong, but in the samples cold rolled with lubricant, the Goss texture was weak and dependent on the heating rate in the annealing step. After researching the effect of cold rolling on the growth of the grains with Goss orientation in silicon steels, Park [13,14] emphasized that with increasing the severity of cold rolling, more distribution of inhibitor precipitates(such as MnSandAlN) is required for control of rapid migration of the grain boundaries in secondary recrystallization. Kim [15] showed that if cold and hot rolling direction is the same, the magnetic induction in the core will greatly increase. Some researchers [16-18] denoted that the strain imposed via cold rolling increases the grain boundary angle and of course high angle grain boundaries have greater speed of migration during annealing.

The purpose of this study was to determine the effect of amount and distribution of cold rolling strain in the transformer core steel after secondary recrystallization.

2. Material and methods
The samples used for cold-rolling were hot-rolled annealed product with chemical composition of 3.4% Si, 0.038% C and 0.1 % Mn. For uniformity of crystal structure and grain size suitable for cold rolling, the samples were annealed at 750 °C for 20 min in argon atmosphere. The annealing furnace was a cubic electrical furnace, with dimensions of 10*10*50 (cm), with Ar gas injection option and the ability to be warmed up to 1300 °C. After annealing, according to the findings of previous researchers [13,14], for better nucleation and development of Goss orientation, the samples were quenched in water with a temperature of 100 °C. For pickling the sheets to remove the surface oxides, sulfuric acid solution (15%) with 0.005 M potassium iodide was used. During the initial cold rolling, all samples were rolled from thickness of 4 mm to 0.4 mm but for investigating the distribution of cold strain in their texture characteristics, three rolling sequences were imposed on them.
All samples of this study were cold strained to 90% in two stages with intermediate annealing between the first and second cold rolling. The rolling machine had two solid cast irons with diameters of 200 mm and length of 500 mm. The purpose of intermediate annealing was reduction of stress and work hardening remaining in the sheets to prevent the surface rupture and better control of texture in the final product. Thus, the samples with two step cold rolling were annealed for 20 min at 850 °C in the electrical furnace under argon gas atmosphere. According to industrial production lines, first recrystallization annealing was applied while carbon removal was performed. The aim of carbon removing was reducing the carbon content of 0.038% to 0.005% by weight because carbon has great influence on the magnetic aging and increases magnetic losses. In the initial annealing step, time should not be so long as to destroy the driving force for secondary recrystallization and growth in the samples. The furnace atmosphere was wethydrogen combined with 20% nitrogen. Annealing was applied at 850°C for 10 min. The washed and degreased sheets were coated with magnesium oxide layer. Final annealing stage is the most crucial manufacturing step that leads to the rapid growth of the Goss grains. Specification of the furnace was like the previous annealing furnace but its atmosphere was dry gas containing 15% hydrogen and the remaining was Nitrogen. The heating rate of 40°C per hour was applied. To consume less protective gas, the samples were left in the furnace 700°C, the furnace was heated to main temperature (1100°C) and the samples were remained for 6 hours in there. In order to investigate the microstructure during the production process, metallographic samples were prepared from thickness (parallel to the rolling direction and rolling surface) at a depth of 0.1 mm below the surface. The samples were mounted, sanded and polished and etched with a solution of 1% Nital for 3 minutes. The graphs were taken with an optical microscope. In this study, texture samples were taken from mid thickness of sheets. After mechanical polishing, the samples were electro polished in a solution containing 800 ml of acetic acid and 200 ml of hydrochloric acid for removal of any remaining effects of mechanical polishing in the surface. Texture measuring was performed by X-ray diffraction and using a Philips X'Pert devise with pole figures of (111), (200), (220) and (311). The pole figure shows the distribution of two-dimensional crystals, such as poles of page (hkl) about the axes of the sample. The Orientation of distribution function was calculated with X'Pert software. At each stage, was used from 4 pole figures to calculate these functions. Each of the texture components has specific position in pole figures and orientation distribution function. Euler angles assume with orthogonal specimens and the crystal axes were shown with three rotations. In the Bunge method, these rotations are $\phi_1$, $\phi$ and $\phi_2$. Measurement of magnetic losses of products was done in a frequency of 0.5 Hz and the magnetic field was around the sheets and done with three Hall sensors. The distance between the poles was 40 mm.

3. Results and Discussion
The importance of cold rolling is in the creation of local in homogeneities such as shear bands and micro bands to create suitable sites for nucleation of the Goss grains. These places lead to the development of the Goss texture during annealing. In the secondary recrystallization, that is the
most important stage of production of electrical sheets, the Goss grains spread with irregular growth process. Much research has been done about the cause of this phenomenon [8-11]. Formerly, it was believed that during cold rolling, the more strain energy is applied on Goss grains and these grains have faster growth in recrystallization. However, today, studies show that the reason of irregular growth of Goss orientation is grain boundaries that are aligned with their surrounding areas. Due to the higher diffusion coefficient, these grain boundaries are less affected by inhibitory properties of precipitates. In fact, after the initial recrystallization, grain boundaries do not reach to the steady state and in the final annealing step the grains that have less inhibitory force in front of their boundaries will have extraordinary growth within the matrix. So, with proper distribution of precipitates that control orientation other Goss can propagate Goss texture. Figure 1 shows the position of the MnS precipitate in grain boundary in sample C2 after the initial annealing.

![Figure 1. Position of MnS precipitate in the grain boundary of sample C2 after the initial annealing](image)

In this case, as seen in Figure 2, as a result of final annealing at 1100 °C in sample A2, the secondary recrystallization structure with average grain size of 3mm has been formed and uncontrolled grain growth can be observed in this case. In this sample, some grains have swallowed the remaining grains that are primary recrystallized grains. Grain boundaries that do not have the ability of rapid growth have been suspended with available inhibitors such as MnS. However, grain growth occurred in two other cases, but the uncontrolled growth of the Goss grains was not so obvious. In sample C2, small grains still exist between the large grains. In Figure 2- c, the annealed structure of this sample is shown. Development of secondary recrystallization in this case results in homogeneous grain growth, and the average size of the grains is 1.5 mm. In this figure, there are no conditions for the uncontrolled growth of the special grains, which can be due to the large number of growing grains as a result of high strain of secondary cold rolling and loss of inhibitory effect of MnS precipitates in the grain boundary that provides the growth condition for most grains.
A precise inspection of all three cases reveals that the size of precipitates is larger compared to earlier stages of production because of the greater suitability of growth conditions due to the long time and high temperature.
Overall, the changes that occur during heating in the final annealing can be divided into three stages: at the first stage, the size of the precipitates prevents the migration of all grains. At the second stage, the temperature increases and the inhibition property of precipitates is reduced. At this point, Goss grains are relieved earlier than other grains due to further diffusion, and find common growth opportunity. At the third stage, by increasing temperature and time, grains with different orientations find opportunity to grow.

By comparing the pole figures and ODFs for the final annealing samples, the intensity of the different components of crystalline texture can be compared. Changing the fibers of $\gamma$ and $\eta$ for different secondary cold rolling strain is shown in Figures 3 and 4.

As can be seen, in sample $A_2$, the intensity of rolling components or gamma fiber is very low, while intensity of Cube and Goss components are significant. The reason for this is favorable conditions in the final annealing for uncontrolled growth of Goss component and annihilating of the rolling components caused by the cold rolling.

![Figure 3](image3.png)

Figure 3. Changes in the intensity of components in the $\gamma$ fiber after final annealing via the amount of strain.

![Figure 4](image4.png)

Figure 4. Changes in the intensity of components in the $\eta$ fiber after final annealing via the amount of strain.

In this sample, the rolling components almost disappear and recrystallization components are very intense. Selective growth of the Goss-oriented grains and disappearance of grains with other
orientation are the main causes of the reduction of rolling components and the increase of recrystallization components.

Texture of sample C2 has the highest intensity component of Goss. Very low components intensity in γ can be seen in sample A2 (Figure 3).

The final annealing of sample A2 almost leads to the disappearance of the γ fiber and for that reason, it is very useful for the development of grains with specific orientation, so that these grains have swallowed the surrounding areas and have changed the dominant orientation (Figures 3-5).

Dependence of η fiber on secondary cold strain is shown in Figure 4. The A2 sample has the maximum intensity of the Goss component—about 20 times of random. The final annealing of sample C2 provides the growing conditions for components other than Goss components and the γ fiber is not weak like the A2 sample.

The phenomenon of secondary recrystallization in grain-oriented steels can be described by two theories: 1-the theory of oriented nucleation and 2-the theory of oriented growth. In theory of oriented nucleation, the grain size is very critical. That is, if a grain has superior size relative to other grains in the fields of primary recrystallization, it can grow uncommonly. In oriented growth theory, two models have been proposed. In the first model, the grain boundaries are low-energy, low-angle, and with high mobility, and in the second model, the grain boundaries are high-energy and high-angle.

The strain imposed in the secondary cold rolling is very impressive at this stage. If this strain is greater, then after the final annealing, the grains will be smaller and more precipitates will be required to control the grain boundary migration. As can be seen in Figure 2, in sample C2, abnormal growth did not occur.

During cold rolling, the Goss grains suffered more strain than other grains, and were thus crystallized earlier than other grains and obtained their original crystallographic orientation. Then these grains occupy neighbors that are high-energy grains and grow.

In this sample, the presence of high strain energy due to the secondary cold rolling significantly increases the number of crystallized nuclei that need space for growing. Of course, with the aggregate interference during growth, their enlargement will stop.

The coherent Goss grain boundaries in the presence of inhibitor precipitates have greater mobility compared to grain boundaries of the other components. In the C2 sample, due to stored strain energy of severe secondary cold rolling, more boundaries are capable for uncommon growth and the presence of precipitates for control of unwanted boundaries is not enough. In sample A2, during the first recrystallization, over much energy of grain boundary has been released and present precipitates can control them, thus Goss grains can have uncommon growth (Figure 5).
Figure 5. Pole figures (PF) of samples obtained after final annealing:
a) SampleA₂ with 80% primary cold rolling and 50% secondary cold rolling
b) SampleB₂ with 70% primary cold rolling and 67% secondary cold rolling
c) SampleC₂ with 50% primary cold rolling and 80% secondary cold rolling

On the other hand, according to the theory of geometrically necessary boundaries (GNBs), during growth of grains, two grains, if placed in contact with the same orientation, will occupy neighbors as a coarse grain. In sample A₂, among the secondary rolling, due to less strain, the possibility of
placing the grains in the same direction is higher and the uncommon growth of the grains can be observed.

The relationship between microstructure and texture and the influence of applied cold strain can be investigated by magnetic losses of obtained steel. In Figure 6, the hysteresis losses in the steel samples with different secondary cold strain can be seen. In sample C2, the magnetic loss is high as can be seen in Figures 2 and 5, in which abnormal growth of the Goss grains occurred.

![Figure 6: Hysteresis losses in the samples after final annealing in terms of secondary cold rolling strain](image)

In other words, the grains which are different from the Goss-oriented grow faster and the main texture of metal is not Goss texture anymore. Therefore, the secondary recrystallization leads to the survival of the major components of \(\gamma\)-fiber and average grain size of 1.5 mm that result in a high magnetic loss.

Decreasing in the hysteresis losses occurred in sample A2 can be seen in Figures 2 to 5. In these figures, Goss grain-oriented (\(<001\>{[110]}\)) in the final annealing have rapid growth and have occupied more areas. However, the average grain size is larger than the other samples and is about 3 mm and it would have a significant impact on reducing the magnetic losses.

4. Conclusion

In this study, three types of cold-rolling were performed on the samples and its importance in the development of texture, microstructure and the hysteresis losses was studied. In one of the cold-rolling processes, primary and secondary strains are 80% and 50%, respectively, and the best conditions for grain growth of Goss orientation during recrystallization were obtained. In another sample, the strains were the same in two stages in which the field was not very favorable for the development of Goss. Applying cold reduction in two steps-50% in the first and 80% in the second stage-resulted in the development of rolling and recrystallization components simultaneously during the final annealing that resulted in more magnetic loss. That was due to the weakening of the inhibitory effect of precipitates and further mobility of the grain boundaries with different orientations during the final annealing. In all three cases, middle annealing resulted in strengthening of the Goss texture and on the other hand, deformation in homogeneities created during cold rolling speeded up the nucleation of this texture at the later annealing stages.
5. References


